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APPENDIX III.

CLINICAL RESULTS

AMA LABORATORIES, INC.

***IN VIVO* SPF AND PPD STUDIES**



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EVALUATION OF SUN PROTECTION
BY SPF DETERMINATION (FDA-TFM)- STATIC

AMA Ref. No.: MS00.SPF.C1168TK.ST20

Date: August 22, 2000

Sponsor: Tri-K Industries
151 Veterans Drive
Northvale, NJ 07647-0128

1.0 Objective:

This panel has been convened to evaluate the effectiveness of a test material as a sunscreen product by determining the Sun Protection Factor (SPF) on human skin in a procedure defined by the Tentative Final Monograph, "Sunscreen Drug Products For Over-The-Counter Human Drugs", (Federal Register Volume 58, Number 90 pages 28194-28302, 1993) using a Xenon arc solar simulator as the UV source.

2.0 Sample Description:

On August 7, 2000 one test sample labeled Sunscreen Lotion EK4-96A was received from Tri-K Industries and assigned AMA Lab No. C-1168.

3.0 Test Material Handling:

Upon arrival at AMA Laboratories, Inc., the test material is assigned a unique laboratory code number and entered into a daily log identifying the lot number, sample description, sponsor, date received and tests requested.

Samples are retained for a period of three months beyond submission of final report unless otherwise specified by the sponsor or if sample is known to be in support of governmental applications, in which case retained samples are kept two years beyond final report submission.

Sample disposition is conducted in compliance with appropriate federal, state and local ordinances.

4.0 Panel Demographics:

Number of subjects enrolled.....	20
Number of subjects completing study.....	20
Age Range.....	18 – 52
Sex.....	Male..... 9
	Female..... 11
Race.....	Caucasian..... 20
	Hispanic..... 0
	Asian..... 0

4.1 Standards For Inclusion In A Study:

- a. Individuals between the ages of 18 and 55.
- b. Individuals free of any dermatological or systemic disorder which would interfere with the results, at the discretion of the Investigator.
- c. Individuals free of any acute or chronic disease that might interfere with or increase the risk of study participation.
- d. Individuals with Fitzpatricks skin type I, II, and III only.
- e. Individuals with no uneven skin tones, pigmentation, scars, other irregularities or hair in test site areas that would interfere with SPF determination.
- f. Individuals who will complete a preliminary medical history form mandated by AMA Laboratories, Inc. and are in general good health.
- g. Individuals who will read, understand and sign an informed consent document relating to the specific type of study they are subscribing. Consent forms are kept on file and are available for examination on the premises of AMA Laboratories, Inc. only.
- h. Individuals able to cooperate with the Investigator and research staff, be willing to have test materials applied according to the protocol, and complete the full course of the study.
- i. Individuals willing to refrain from using any sunscreen products, sunbathing or tanning bed use, 24 hours prior to study initiation and the entire duration of the study.

- j. Individuals with excessive hair on their back who are willing to clip or shave their hair.

4.2 Standards Of Exclusion From The Study:

- a. Individuals who are under doctor's care.
- b. Individuals who are currently taking any medication (topical or systemic) that may mask or interfere with the test results.
- c. Subjects with a history of any form of skin cancer, melanoma, lupus, psoriasis, connective tissue disease, diabetes or any disease that would increase the risk associated with study participation.
- d. Individuals diagnosed with chronic skin allergies.
- e. Individuals with a history of adverse effects upon sun exposure.
- f. Female volunteers who indicate that they are pregnant or nursing.
- g. Individuals with blemishes, nevi, sunburn, suntan, scars, moles, active dermal lesions or uneven pigmentation in the test sites.
- h. Individuals with known hypersensitivity to any sunscreen products.

4.3 Informed Consent And Medical History Forms:

An informed consent was obtained from each volunteer prior to initiating the study describing reasons for the study, possible adverse effects, associated risks and potential benefits of the treatment and their limits of liability. Panelists signed and dated the informed consent document to indicate their authorization to proceed and acknowledge their understanding of the contents. Each subject was assigned a permanent identification number and completed an extensive medical history form. These forms along with the signed consent forms are available for inspection on the premises of AMA Laboratories, Inc. only. Reference 21 CFR Ch. 1 Part 50, Subpart B.

A trained technician performed a physical examination of the panelist's back to determine if study eligibility criteria were satisfied.

4.4 Panel Composition:

Healthy volunteers over the age of 18 years were recruited for this study. The panel consisted of fair-skin individuals with skin types I, II or III defined as follows (Federal Register Vol. 58, No. 90: 28299, 1993).*

Type I - Always burns easily; never tans

Type II - Always burns easily; tans minimally

Type III - Burns moderately; tans gradually

* - Based on the first 30 to 45 minutes sun exposure after a winter season of no sun exposure.

4.5 Institutional Review Board:

Reference: CFR Title 21 Part 56, Subparts A, B, C, and D. The IRB of AMA Laboratories, Inc., consists of five or more individuals, chosen from within the company for technical expertise and from the local community for lay interaction. The list of IRB members is kept on file at AMA Laboratories, Inc. and is available for inspection during the hours of operation.

5.0 Artificial Light Source:

The light source employed is a 150 watt Xenon Arc Solar Simulator (Solar Light Co., Philadelphia, Pennsylvania, Model 12S, Model 14S or Model 600) having a continuous emission spectrum in the UVB range from 290 to 320 nm. Xenon arc is selected on the basis of its black body radiation temperature of 6000° K which produces continuous UV spectra (all wavelengths) substantially equivalent to that of natural sunlight¹.

This device is equipped with a dichroic mirror (which reflects all radiation below 400nm) and works in conjunction with a 1 mm thick Schott WG-320 filter (which absorbs all radiation below 290nm) to produce simulation of the solar UVA-UVB spectrum. A 1 mm thick UG 11 filter (black lens) was added to remove reflected (infra-red, greater than 700 nm) heat and remaining visible radiation. UVB radiation was monitored continuously during exposure using a Model DCS-1 Sunburn UV Meter/Dose Controller System (Solar Light Co.) formerly known as the Robertson-Berger Sunburn Meter

(R-B meter). Measurements were taken at a position within 8 mm from the surface of the skin. The field of irradiation was 1 cm in diameter. The solar simulator was allowed a warm up time of at least 15 minutes before use and power supply output was recorded.

Realignment of the Light Sources and calibration of the sunburn meters are conducted semi-annually by independent certification facilities and more often as necessary at the discretion of the operating technician or Investigator.

1/ Berger, D.S.: Specification and Design of Solar Ultraviolet Simulators. J. Invest. Dermatol. 53: 192-199, 1969.

6.0 Procedure:

STATIC SPF DETERMINATION (INCLUDING 8% HOMOSALATE STANDARD)

The infrascapular area of the back to the right and left of the midline was used. Within this area, 50 cm² rectangular test sites were delineated with a gentian violet surgical skin marker. Sites were observed to ensure uniform pigmentation, skin tone and texture, and absence of warts, moles, nevi, scars, blemishes and active dermal lesions. Any areas that might be expected to produce erratic results were not used for UV exposures.

The procedure for this study is outlined in the Federal Register, Vol. 58: No.90, 28194-28302. One test site area served to determine each subject's Minimal Erythema Dose (MED). A minimum of five UV exposures was administered within this site. The individual subject's MED is the shortest time of exposure that produces minimally perceptible erythema at 22 to 24 hours post irradiation.

The test material and 8% homosalate standard were shaken and/or swirled with a glass rod before use and were evenly applied using plastic volumetric syringes to a rectangular areas measuring 5 cm x 10 cm (50 cm²) for a final concentration of 2.0 mg/cm². Evenness of application was verified by observation with a Woods Lamp.

Fifteen minutes after application, a protected site received a series of seven UV exposures based upon previously determined MED.

The UV exposures for test products and 8% homosalate standard were calculated from previously determined MED and the intended SPF as follows:

SPF 4: MED times 0.64x, 0.80x, 0.90x, 1.00x, 1.10x, 1.25x and 1.56x

SPF 10: MED times 0.69x, 0.83x, 0.91x, 1.00x, 1.09x, 1.20x and 1.44x where x equals the expected SPF of the product.

7.0 Evaluation Of Responses:

Twenty-two to twenty-four hours post exposure, the subjects are instructed to return to the testing facility for evaluation of delayed erythemic responses. The technician who evaluates the MED did not know the identity of the test product application sites and UV exposures. Also he/she was not the same person to have applied the sunscreen product to the test site or administered the doses of UV radiation.

$$\text{SPF} = \frac{\text{Protected MED}}{\text{Final unprotected MED}}$$

Visual grading scale:

- 0 = No Erythema
- ? = Questionable Erythema
- 1 = Minimal Erythema
- 2 = Slight Erythema
- 3 = Well-Defined Erythema
- 4 = Erythema and Edema
- 5 = Erythema and Edema in vesicles

- 7.1 All technical employees of AMA Laboratories, Inc. are required to take and pass a visual discrimination examination conducted by a Board Certified Ophthalmologist using the Farnsworth-Munsell 100 Hue Test as published; which determines a person's ability to discern color against a black background. This test was additionally modified to include a flesh tone background more nearly approaching actual use conditions, wherein erythematous skin is graded according to intensity.

8.0 Determination of the Test Product's SPF Value and PCD:

Calculation of SPF and PCD – The mean SPF value (x) was calculated. The standard deviation was determined (s). The upper 5% point was obtained from the t distribution table with n-1 degrees of freedom (t). First, A was calculated as follows:

$$A = \frac{(t)(s)}{\sqrt{n}}$$

Therefore, the label SPF for panels using a minimum of 20 evaluable subjects is the largest whole number less than the mean SPF minus A.

$$\text{Label SPF} = \text{Mean SPF} - A$$

The Product Category Designation (PCD), for labeling purposes, was assigned based on the mean SPF and PCD ranking according to the TFM. Classifications may be Ultra High, Very High, High, Moderate or Minimal.

9.0 Rejection Criteria:

Panelist's results were rejected and the panelist replaced if:

1. An exposure series fails to elicit an MED response on the untreated skin. The test is considered a technical failure even if the MED response is observed in the protected site.
2. The responses on the protected area are randomly absent, indicating uneven product spreading, non-constant light irradiance or unstable product.
3. All exposures in a series elicit responses - thus prohibiting any MED calculation.

10.0 Results: Please see attached Table.

11.0 Observations:

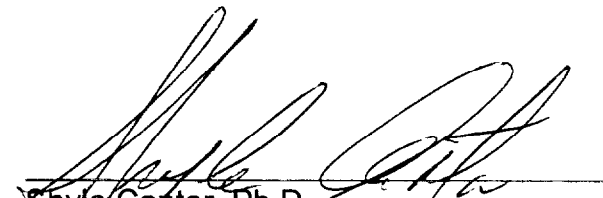
No adverse effects or unexpected reactions of any kind were observed on any of the subjects.

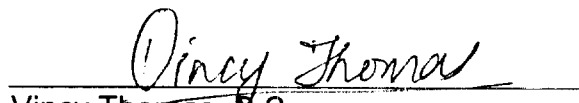
12.0 Archiving:

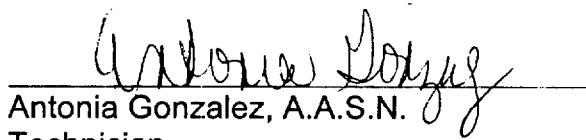
All original samples, raw data sheets, technicians notebooks, correspondence files and copies of final reports and remaining specimens are maintained on premises of AMA Laboratories, Inc. in limited access storage files marked "Archive". A duplicate disk copy of final reports is separately archived in a bank safe deposit vault.

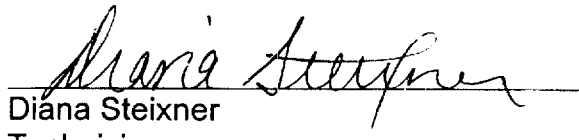
13.0 Conclusions:

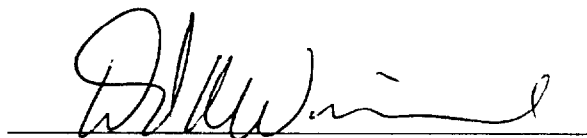
The Sun Protection Factor (SPF) of the above test material (AMA Lab No.: C-1168; Client No.: Sunscreen Lotion EK4-96A) when tested on twenty subject as described herein under static conditions yielded the mean SPF value of 10.00, which can be assigned a Products Category Designation (PCD) of High according to the reference. The mean SPF of the 8% homosalate standard on the same panel was 4.35

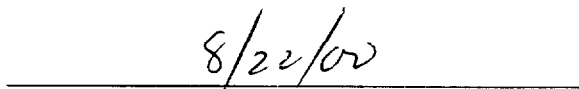

Shyla Cantor, Ph.D.
Study Director


Vincy Thomas, B.S.
Technician


Antonia Gonzalez, A.A.S.N.
Technician


Diana Steixner
Technician


David R. Winne, B.S.
Quality Assurance Supervisor


Date

Table

Sponsor: Tri-K Industries
 AMA Lab No.: C-1168
 Client No.: Sunscreen Lotion EK4-96A

Subject ID #	Sex	MED/HR	I (Amps)	Skin Type	MED I J/M ²	MED II J/M ²	STD (8% HMS)	SPF Value
36 2460	F	126.7	8.0	I	46.20	46.20	4.40	9.10
36 3821	M	127.0	7.0	II	60.89	60.89	4.40	9.10
64 1135	M	125.6	8.1	II	75.59	75.59	4.40	9.00
68 6947	M	128.3	9.0	II	60.89	60.89	4.40	10.90
60 0476	F	126.0	7.5	I	46.20	46.20	4.00	10.90
40 6694	F	129.5	7.0	II	60.89	60.89	4.00	10.90
40 3407	F	126.6	8.1	I	46.20	46.20	4.40	10.00
38 4338	F	126.4	8.7	I	46.20	46.20	4.40	10.00
44 2511	F	130.0	8.0	I	46.20	46.20	4.00	10.90
94 7230	F	125.7	8.0	I	46.20	46.20	4.40	10.00
66 8507	M	127.8	8.0	II	75.59	75.59	4.40	10.90
50 4079	M	125.5	8.6	II	60.89	60.89	5.00	9.10
54 3185	M	125.0	8.0	I	46.20	46.20	4.40	10.90
62 5824	M	125.8	7.8	II	60.89	60.89	4.40	10.90
82 4044	M	127.4	7.8	II	60.89	60.89	4.40	9.10
66 4731	F	127.0	8.2	I	46.20	46.20	4.40	10.90
52 4724	F	128.0	8.0	I	46.20	46.20	4.00	9.10
60 3008	M	127.0	8.1	I	46.20	46.20	4.40	9.10
50 1729	F	125.0	8.0	I	46.20	46.20	4.40	9.10
46 9704	F	127.4	6.0	II	60.89	60.89	4.40	10.00
MEAN (x)							4.35	10.00
STANDARD DEV (s)							0.22	0.83
STD. ERROR							0.05	0.19
S.E. % OF MEAN							1.15	1.90
N							20	20
UPPER 5% t DIST.							2.0930	1.7291
A VALUES							1.0E-01	3.2E-01
LABEL SPF							4	10

MED: Minimal Erythral Dose
 I: Intensity of light source

Evaluation Period:

This study was conducted from August 7, 2000
 through August 22, 2000.



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EVALUATION OF SUN PROTECTION
BY SPF DETERMINATION (FDA-TFM)- STATIC

AMA Ref. No.: MS00.SPF.C1169TK.ST20

Date: August 22, 2000

Sponsor: Tri-K Industries
151 Veterans Drive
Northvale, NJ 07647-0128

1.0 Objective:

This panel has been convened to evaluate the effectiveness of a test material as a sunscreen product by determining the Sun Protection Factor (SPF) on human skin in a procedure defined by the Tentative Final Monograph, "Sunscreen Drug Products For Over-The-Counter Human Drugs", (Federal Register Volume 58, Number 90 pages 28194-28302, 1993) using a Xenon arc solar simulator as the UV source.

2.0 Sample Description:

On August 7, 2000 one test sample labeled Sunscreen Lotion EK4-96C was received from Tri-K Industries and assigned AMA Lab No. C-1169.

3.0 Test Material Handling:

Upon arrival at AMA Laboratories, Inc., the test material is assigned a unique laboratory code number and entered into a daily log identifying the lot number, sample description, sponsor, date received and tests requested.

Samples are retained for a period of three months beyond submission of final report unless otherwise specified by the sponsor or if sample is known to be in support of governmental applications, in which case retained samples are kept two years beyond final report submission.

Sample disposition is conducted in compliance with appropriate federal, state and local ordinances.

4.0 Panel Demographics:

Number of subjects enrolled.....	20
Number of subjects completing study.....	20
Age Range.....	18 – 52
Sex.....	Male..... 9
	Female..... 11
Race.....	Caucasian..... 20
	Hispanic..... 0
	Asian..... 0

4.1 Standards For Inclusion In A Study:

- a. Individuals between the ages of 18 and 55.
- b. Individuals free of any dermatological or systemic disorder which would interfere with the results, at the discretion of the Investigator.
- c. Individuals free of any acute or chronic disease that might interfere with or increase the risk of study participation.
- d. Individuals with Fitzpatrick's skin type I, II, and III only.
- e. Individuals with no uneven skin tones, pigmentation, scars, other irregularities or hair in test site areas that would interfere with SPF determination.
- f. Individuals who will complete a preliminary medical history form mandated by AMA Laboratories, Inc. and are in general good health.
- g. Individuals who will read, understand and sign an informed consent document relating to the specific type of study they are subscribing. Consent forms are kept on file and are available for examination on the premises of AMA Laboratories, Inc. only.
- h. Individuals able to cooperate with the Investigator and research staff, be willing to have test materials applied according to the protocol, and complete the full course of the study.
- i. Individuals willing to refrain from using any sunscreen products, sunbathing or tanning bed use, 24 hours prior to study initiation and the entire duration of the study.

- j. Individuals with excessive hair on their back who are willing to clip or shave their hair.

4.2 Standards Of Exclusion From The Study:

- a. Individuals who are under doctor's care.
- b. Individuals who are currently taking any medication (topical or systemic) that may mask or interfere with the test results.
- c. Subjects with a history of any form of skin cancer, melanoma, lupus, psoriasis, connective tissue disease, diabetes or any disease that would increase the risk associated with study participation.
- d. Individuals diagnosed with chronic skin allergies.
- e. Individuals with a history of adverse effects upon sun exposure.
- f. Female volunteers who indicate that they are pregnant or nursing.
- g. Individuals with blemishes, nevi, sunburn, suntan, scars, moles, active dermal lesions or uneven pigmentation in the test sites.
- h. Individuals with known hypersensitivity to any sunscreen products.

4.3 Informed Consent And Medical History Forms:

An informed consent was obtained from each volunteer prior to initiating the study describing reasons for the study, possible adverse effects, associated risks and potential benefits of the treatment and their limits of liability. Panelists signed and dated the informed consent document to indicate their authorization to proceed and acknowledge their understanding of the contents. Each subject was assigned a permanent identification number and completed an extensive medical history form. These forms along with the signed consent forms are available for inspection on the premises of AMA Laboratories, Inc. only. Reference 21 CFR Ch. 1 Part 50, Subpart B.

A trained technician performed a physical examination of the panelist's back to determine if study eligibility criteria were satisfied.

4.4 Panel Composition:

Healthy volunteers over the age of 18 years were recruited for this study. The panel consisted of fair-skin individuals with skin types I, II or III defined as follows (Federal Register Vol. 58, No. 90: 28299, 1993).*

Type I - Always burns easily; never tans

Type II - Always burns easily; tans minimally

Type III - Burns moderately; tans gradually

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4.5 Institutional Review Board:

Reference: CFR Title 21 Part 56, Subparts A, B, C, and D. The IRB of AMA Laboratories, Inc., consists of five or more individuals, chosen from within the company for technical expertise and from the local community for lay interaction. The list of IRB members is kept on file at AMA Laboratories, Inc. and is available for inspection during the hours of operation.

5.0 Artificial Light Source:

The light source employed is a 150 watt Xenon Arc Solar Simulator (Solar Light Co., Philadelphia, Pennsylvania, Model 12S, Model 14S or Model 600) having a continuous emission spectrum in the UVB range from 290 to 320 nm. Xenon arc is selected on the basis of its black body radiation temperature of 6000° K which produces continuous UV spectra (all wavelengths) substantially equivalent to that of natural sunlight¹.

This device is equipped with a dichroic mirror (which reflects all radiation below 400nm) and works in conjunction with a 1 mm thick Schott WG-320 filter (which absorbs all radiation below 290nm) to produce simulation of the solar UVA-UVB spectrum. A 1 mm thick UG 11 filter (black lens) was added to remove reflected (infra-red, greater than 700 nm) heat and remaining visible radiation. UVB radiation was monitored continuously during exposure using a Model DCS-1 Sunburn UV Meter/Dose Controller System (Solar Light Co.) formerly known as the Robertson-Berger Sunburn Meter

(R-B meter). Measurements were taken at a position within 8 mm from the surface of the skin. The field of irradiation was 1 cm in diameter. The solar simulator was allowed a warm up time of at least 15 minutes before use and power supply output was recorded.

Realignment of the Light Sources and calibration of the sunburn meters are conducted semi-annually by independent certification facilities and more often as necessary at the discretion of the operating technician or Investigator.

1/ Berger, D.S.: Specification and Design of Solar Ultraviolet Simulators. J. Invest. Dermatol. 53: 192-199, 1969.

6.0 Procedure:

STATIC SPF DETERMINATION (INCLUDING 8% HOMOSALATE STANDARD)

The infrascapular area of the back to the right and left of the midline was used. Within this area, 50 cm² rectangular test sites were delineated with a gentian violet surgical skin marker. Sites were observed to ensure uniform pigmentation, skin tone and texture, and absence of warts, moles, nevi, scars, blemishes and active dermal lesions. Any areas that might be expected to produce erratic results were not used for UV exposures.

The procedure for this study is outlined in the Federal Register, Vol. 58: No.90, 28194-28302. One test site area served to determine each subject's Minimal Erythema Dose (MED). A minimum of five UV exposures was administered within this site. The individual subject's MED is the shortest time of exposure that produces minimally perceptible erythema at 22 to 24 hours post irradiation.

The test material and 8% homosalate standard were shaken and/or swirled with a glass rod before use and were evenly applied using plastic volumetric syringes to a rectangular areas measuring 5 cm x 10 cm (50 cm²) for a final concentration of 2.0 mg/cm². Evenness of application was verified by observation with a Woods Lamp.

Fifteen minutes after application, a protected site received a series of seven UV exposures based upon previously determined MED.

The UV exposures for test products and 8% homosalate standard were calculated from previously determined MED and the intended SPF as follows:

SPF 4: MED times 0.64x, 0.80x, 0.90x, 1.00x, 1.10x, 1.25x and 1.56x

SPF 15: MED times 0.76x, 0.87x, 0.93x, 1.00x, 1.07x, 1.15x and 1.32x where x equals the expected SPF of the product.

7.0 Evaluation Of Responses:

Twenty-two to twenty-four hours post exposure, the subjects are instructed to return to the testing facility for evaluation of delayed erythemic responses. The technician who evaluates the MED did not know the identity of the test product application sites and UV exposures. Also he/she was not the same person to have applied the sunscreen product to the test site or administered the doses of UV radiation.

$$\text{SPF} = \frac{\text{Protected MED}}{\text{Final unprotected MED}}$$

Visual grading scale:

- 0 = No Erythema
- ? = Questionable Erythema
- 1 = Minimal Erythema
- 2 = Slight Erythema
- 3 = Well-Defined Erythema
- 4 = Erythema and Edema
- 5 = Erythema and Edema in vesicles

- 7.1 All technical employees of AMA Laboratories, Inc. are required to take and pass a visual discrimination examination conducted by a Board Certified Ophthalmologist using the Farnsworth-Munsell 100 Hue Test as published; which determines a person's ability to discern color against a black background. This test was additionally modified to include a flesh tone background more nearly approaching actual use conditions, wherein erythematous skin is graded according to intensity.

8.0 Determination of the Test Product's SPF Value and PCD:

Calculation of SPF and PCD – The mean SPF value (x) was calculated. The standard deviation was determined (s). The upper 5% point was obtained from the t distribution table with n-1 degrees of freedom (t). First, A was calculated as follows:

$$A = \frac{(t)(s)}{\sqrt{n}}$$

Therefore, the label SPF for panels using a minimum of 20 evaluable subjects is the largest whole number less than the mean SPF minus A.

$$\text{Label SPF} = \text{Mean SPF} - A$$

The Product Category Designation (PCD), for labeling purposes, was assigned based on the mean SPF and PCD ranking according to the TFM. Classifications may be Ultra High, Very High, High, Moderate or Minimal.

9.0 Rejection Criteria:

Panelist's results were rejected and the panelist replaced if:

1. An exposure series fails to elicit an MED response on the untreated skin. The test is considered a technical failure even if the MED response is observed in the protected site.
2. The responses on the protected area are randomly absent, indicating uneven product spreading, non-constant light irradiance or unstable product.
3. All exposures in a series elicit responses - thus prohibiting any MED calculation.

10.0 Results: Please see attached Table.

11.0 Observations:

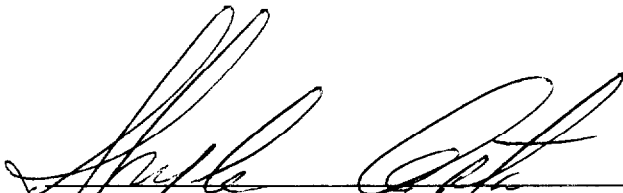
No adverse effects or unexpected reactions of any kind were observed on any of the subjects.

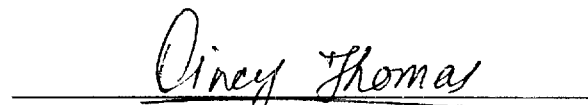
12.0 Archiving:

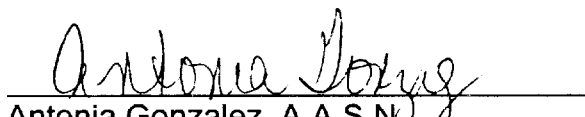
All original samples, raw data sheets, technicians notebooks, correspondence files and copies of final reports and remaining specimens are maintained on premises of AMA Laboratories, Inc. in limited access storage files marked "Archive". A duplicate disk copy of final reports is separately archived in a bank safe deposit vault.

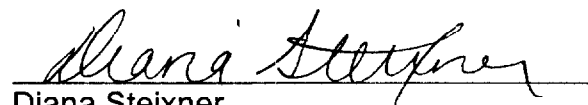
13.0 Conclusions:

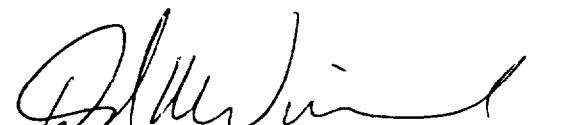
The Sun Protection Factor (SPF) of the above test material (AMA Lab No.: C-1169; Client No.: Sunscreen Lotion EK4-96C) when tested on twenty subject as described herein under static conditions yielded the mean SPF value of 18.41, which can be assigned a Products Category Designation (PCD) of Very High according to the reference. The mean SPF of the 8% homosalate standard on the same panel was 4.35



Shyla Cantor, Ph.D.
Study Director


Vincy Thomas, B.S.
Technician


Antonia Gonzalez, A.A.S.N.
Technician


Diana Steixner
Technician


David R. Winne, B.S.
Quality Assurance Supervisor


Date

Table

Sponsor: Tri-K Industries
 AMA Lab No.: C-1169
 Client No.: Sunscreen Lotion EK4-96C

Subject ID #	Sex	MED/HR	I (Amps)	Skin Type	MED I J/M ²	MED II J/M ²	STD (8% HMS)	SPF Value
36 2460	F	126.7	8.0	I	46.20	46.20	4.40	16.35
36 3821	M	127.0	7.0	II	60.89	60.89	4.40	16.35
64 1135	M	125.6	8.1	II	75.59	75.59	4.40	21.60
68 6947	M	128.3	9.0	II	60.89	60.89	4.40	18.00
60 0476	F	126.0	7.5	I	46.20	46.20	4.00	18.00
40 6694	F	129.5	7.0	II	60.89	60.89	4.00	18.00
40 3407	F	126.6	8.1	I	46.20	46.20	4.40	21.60
38 4338	F	126.4	8.7	I	46.20	46.20	4.40	21.60
44 2511	F	130.0	8.0	I	46.20	46.20	4.00	18.00
94 7230	F	125.7	8.0	I	46.20	46.20	4.40	21.60
66 8507	M	127.8	8.0	II	75.59	75.59	4.40	18.00
50 4079	M	125.5	8.6	II	60.89	60.89	5.00	16.35
54 3185	M	125.0	8.0	I	46.20	46.20	4.40	16.35
62 5824	M	125.8	7.8	II	60.89	60.89	4.40	18.00
82 4044	M	127.4	7.8	II	60.89	60.89	4.40	21.60
66 4731	F	127.0	8.2	I	46.20	46.20	4.40	18.00
52 4724	F	128.0	8.0	I	46.20	46.20	4.00	16.35
60 3008	M	127.0	8.1	I	46.20	46.20	4.40	16.35
50 1729	F	125.0	8.0	I	46.20	46.20	4.40	18.00
46 9704	F	127.4	6.0	II	60.89	60.89	4.40	18.00
MEAN (x)							4.35	18.41
STANDARD DEV (s)							0.22	2.02
STD. ERROR							0.05	0.45
S.E. % OF MEAN							1.15	2.44
N							20	20
UPPER 5% t DIST.							2.0930	1.7291
A VALUES							1.0E-01	7.8E-01
LABEL SPF							4	18

MED: Minimal Erythema Dose
 I: Intensity of light source

Evaluation Period:

This study was conducted from August 7, 2000
 through August 22, 2000.



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New City, NY 10956 USA
(914) 634-4300
FAX: (914) 638-4872

EVALUATION OF SUN PROTECTION
BY SPF DETERMINATION (FDA-TFM)- STATIC

AMA Ref. No.: MS00.SPF.C1170TK.ST20

Date: August 22, 2000

Sponsor: Tri-K Industries
151 Veterans Drive
Northvale, NJ 07647-0128

1.0 Objective:

This panel has been convened to evaluate the effectiveness of a test material as a sunscreen product by determining the Sun Protection Factor (SPF) on human skin in a procedure defined by the Tentative Final Monograph, "Sunscreen Drug Products For Over-The-Counter Human Drugs", (Federal Register Volume 58, Number 90 pages 28194-28302, 1993) using a Xenon arc solar simulator as the UV source.

2.0 Sample Description:

On August 7, 2000 one test sample labeled Sunscreen Lotion EK4-95E was received from Tri-K Industries and assigned AMA Lab No. C-1170.

3.0 Test Material Handling:

Upon arrival at AMA Laboratories, Inc., the test material is assigned a unique laboratory code number and entered into a daily log identifying the lot number, sample description, sponsor, date received and tests requested.

Samples are retained for a period of three months beyond submission of final report unless otherwise specified by the sponsor or if sample is known to be in support of governmental applications, in which case retained samples are kept two years beyond final report submission.

Sample disposition is conducted in compliance with appropriate federal, state and local ordinances.

4.0 Panel Demographics:

Number of subjects enrolled.....	20
Number of subjects completing study.....	20
Age Range.....	18 – 52
Sex.....	Male..... 9
	Female..... 11
Race.....	Caucasian..... 20
	Hispanic..... 0
	Asian..... 0

4.1 Standards For Inclusion In A Study:

- a. Individuals between the ages of 18 and 55.
- b. Individuals free of any dermatological or systemic disorder which would interfere with the results, at the discretion of the Investigator.
- c. Individuals free of any acute or chronic disease that might interfere with or increase the risk of study participation.
- d. Individuals with Fitzpatrick's skin type I, II, and III only.
- e. Individuals with no uneven skin tones, pigmentation, scars, other irregularities or hair in test site areas that would interfere with SPF determination.
- f. Individuals who will complete a preliminary medical history form mandated by AMA Laboratories, Inc. and are in general good health.
- g. Individuals who will read, understand and sign an informed consent document relating to the specific type of study they are subscribing. Consent forms are kept on file and are available for examination on the premises of AMA Laboratories, Inc. only.
- h. Individuals able to cooperate with the Investigator and research staff, be willing to have test materials applied according to the protocol, and complete the full course of the study.
- i. Individuals willing to refrain from using any sunscreen products, sunbathing or tanning bed use, 24 hours prior to study initiation and the entire duration of the study.

- j. Individuals with excessive hair on their back who are willing to clip or shave their hair.

4.2 Standards Of Exclusion From The Study:

- a. Individuals who are under doctor's care.
- b. Individuals who are currently taking any medication (topical or systemic) that may mask or interfere with the test results.
- c. Subjects with a history of any form of skin cancer, melanoma, lupus, psoriasis, connective tissue disease, diabetes or any disease that would increase the risk associated with study participation.
- d. Individuals diagnosed with chronic skin allergies.
- e. Individuals with a history of adverse effects upon sun exposure.
- f. Female volunteers who indicate that they are pregnant or nursing.
- g. Individuals with blemishes, nevi, sunburn, suntan, scars, moles, active dermal lesions or uneven pigmentation in the test sites.
- h. Individuals with known hypersensitivity to any sunscreen products.

4.3 Informed Consent And Medical History Forms:

An informed consent was obtained from each volunteer prior to initiating the study describing reasons for the study, possible adverse effects, associated risks and potential benefits of the treatment and their limits of liability. Panelists signed and dated the informed consent document to indicate their authorization to proceed and acknowledge their understanding of the contents. Each subject was assigned a permanent identification number and completed an extensive medical history form. These forms along with the signed consent forms are available for inspection on the premises of AMA Laboratories, Inc. only. Reference 21 CFR Ch. 1 Part 50, Subpart B.

A trained technician performed a physical examination of the panelist's back to determine if study eligibility criteria were satisfied.

4.4 Panel Composition:

Healthy volunteers over the age of 18 years were recruited for this study. The panel consisted of fair-skin individuals with skin types I, II or III defined as follows (Federal Register Vol. 58, No. 90: 28299, 1993).*

Type I - Always burns easily; never tans

Type II - Always burns easily; tans minimally

Type III - Burns moderately; tans gradually

* - Based on the first 30 to 45 minutes sun exposure after a winter season of no sun exposure.

4.5 Institutional Review Board:

Reference: CFR Title 21 Part 56, Subparts A, B, C, and D. The IRB of AMA Laboratories, Inc., consists of five or more individuals, chosen from within the company for technical expertise and from the local community for lay interaction. The list of IRB members is kept on file at AMA Laboratories, Inc. and is available for inspection during the hours of operation.

5.0 Artificial Light Source:

The light source employed is a 150 watt Xenon Arc Solar Simulator (Solar Light Co., Philadelphia, Pennsylvania, Model 12S, Model 14S or Model 600) having a continuous emission spectrum in the UVB range from 290 to 320 nm. Xenon arc is selected on the basis of its black body radiation temperature of 6000° K which produces continuous UV spectra (all wavelengths) substantially equivalent to that of natural sunlight¹.

This device is equipped with a dichroic mirror (which reflects all radiation below 400nm) and works in conjunction with a 1 mm thick Schott WG-320 filter (which absorbs all radiation below 290nm) to produce simulation of the solar UVA-UVB spectrum. A 1 mm thick UG 11 filter (black lens) was added to remove reflected (infra-red, greater than 700 nm) heat and remaining visible radiation. UVB radiation was monitored continuously during exposure using a Model DCS-1 Sunburn UV Meter/Dose Controller System (Solar Light Co.) formerly known as the Robertson-Berger Sunburn Meter

(R-B meter). Measurements were taken at a position within 8 mm from the surface of the skin. The field of irradiation was 1 cm in diameter. The solar simulator was allowed a warm up time of at least 15 minutes before use and power supply output was recorded.

Realignment of the Light Sources and calibration of the sunburn meters are conducted semi-annually by independent certification facilities and more often as necessary at the discretion of the operating technician or Investigator.

1/ Berger, D.S.: Specification and Design of Solar Ultraviolet Simulators. J. Invest. Dermatol. 53: 192-199, 1969.

6.0 Procedure:

STATIC SPF DETERMINATION (INCLUDING 8% HOMOSALATE STANDARD)

The infrascapular area of the back to the right and left of the midline was used. Within this area, 50 cm² rectangular test sites were delineated with a gentian violet surgical skin marker. Sites were observed to ensure uniform pigmentation, skin tone and texture, and absence of warts, moles, nevi, scars, blemishes and active dermal lesions. Any areas that might be expected to produce erratic results were not used for UV exposures.

The procedure for this study is outlined in the Federal Register, Vol. 58: No.90, 28194-28302. One test site area served to determine each subject's Minimal Erythema Dose (MED). A minimum of five UV exposures was administered within this site. The individual subject's MED is the shortest time of exposure that produces minimally perceptible erythema at 22 to 24 hours post irradiation.

The test material and 8% homosalate standard were shaken and/or swirled with a glass rod before use and were evenly applied using plastic volumetric syringes to a rectangular areas measuring 5 cm x 10 cm (50 cm²) for a final concentration of 2.0 mg/cm². Evenness of application was verified by observation with a Woods Lamp.

Fifteen minutes after application, a protected site received a series of seven UV exposures based upon previously determined MED.

The UV exposures for test products and 8% homosalate standard were calculated from previously determined MED and the intended SPF as follows:

SPF 4: MED times 0.64x, 0.80x, 0.90x, 1.00x, 1.10x, 1.25x and 1.56x

SPF 10: MED times 0.69x, 0.83x, 0.91x, 1.00x, 1.09x, 1.20x and 1.44x where x equals the expected SPF of the product.

7.0 Evaluation Of Responses:

Twenty-two to twenty-four hours post exposure, the subjects are instructed to return to the testing facility for evaluation of delayed erythemic responses. The technician who evaluates the MED did not know the identity of the test product application sites and UV exposures. Also he/she was not the same person to have applied the sunscreen product to the test site or administered the doses of UV radiation.

$$\text{SPF} = \frac{\text{Protected MED}}{\text{Final unprotected MED}}$$

Visual grading scale:

- 0 = No Erythema
- ? = Questionable Erythema
- 1 = Minimal Erythema
- 2 = Slight Erythema
- 3 = Well-Defined Erythema
- 4 = Erythema and Edema
- 5 = Erythema and Edema in vesicles

- 7.1 All technical employees of AMA Laboratories, Inc. are required to take and pass a visual discrimination examination conducted by a Board Certified Ophthalmologist using the Farnsworth-Munsell 100 Hue Test as published; which determines a person's ability to discern color against a black background. This test was additionally modified to include a flesh tone background more nearly approaching actual use conditions, wherein erythematous skin is graded according to intensity.

8.0 Determination of the Test Product's SPF Value and PCD:

Calculation of SPF and PCD – The mean SPF value (x) was calculated. The standard deviation was determined (s). The upper 5% point was obtained from the t distribution table with n-1 degrees of freedom (t). First, A was calculated as follows:

$$A = \frac{(t)(s)}{\sqrt{n}}$$

Therefore, the label SPF for panels using a minimum of 20 evaluable subjects is the largest whole number less than the mean SPF minus A.

$$\text{Label SPF} = \text{Mean SPF} - A$$

The Product Category Designation (PCD), for labeling purposes, was assigned based on the mean SPF and PCD ranking according to the TFM. Classifications may be Ultra High, Very High, High, Moderate or Minimal.

9.0 Rejection Criteria:

Panelist's results were rejected and the panelist replaced if:

1. An exposure series fails to elicit an MED response on the untreated skin. The test is considered a technical failure even if the MED response is observed in the protected site.
2. The responses on the protected area are randomly absent, indicating uneven product spreading, non-constant light irradiance or unstable product.
3. All exposures in a series elicit responses - thus prohibiting any MED calculation.

10.0 Results: Please see attached Table.

11.0 Observations:


No adverse effects or unexpected reactions of any kind were observed on any of the subjects.

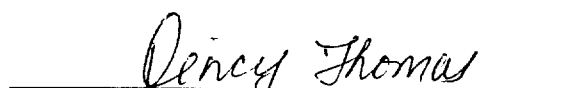
12.0 Archiving:

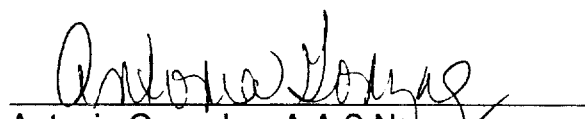
All original samples, raw data sheets, technicians notebooks, correspondence files and copies of final reports and remaining specimens are maintained on premises of AMA Laboratories, Inc. in limited access storage files marked "Archive". A duplicate disk copy of final reports is separately archived in a bank safe deposit vault.

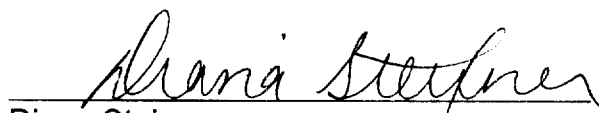
13.0 Conclusions:

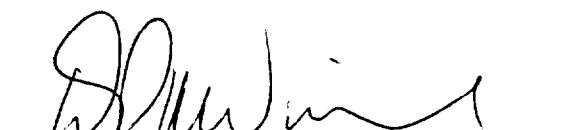
The Sun Protection Factor (SPF) of the above test material (AMA Lab No.: C-1170; Client No.: Sunscreen Lotion EK4-95E) when tested on twenty subject as described herein under static conditions yielded the mean SPF value of 10.76, which can be assigned a Products Category Designation (PCD) of High according to the reference. The mean SPF of the 8% homosalate standard on the same panel was 4.35



Shyla Cantor, Ph.D.
Study Director


Vincy Thomas, B.S.
Technician


Antonia Gonzalez, A.A.S.N.
Technician


Diana Steixner
Technician


David R. Winne, B.S.
Quality Assurance Supervisor


Date

Table

Sponsor: Tri-K Industries
 AMA Lab No.: C-1170
 Client No.: Sunscreen Lotion EK4-95E

Subject ID #	Sex	MED/HR	I (Amps)	Skin Type	MED I J/M ²	MED II J/M ²	STD (8% HMS)	SPF Value
36 2460	F	126.7	8.0	I	46.20	46.20	4.40	10.90
36 3821	M	127.0	7.0	II	60.89	60.89	4.40	10.00
64 1135	M	125.6	8.1	II	75.59	75.59	4.40	10.90
68 6947	M	128.3	9.0	II	60.89	60.89	4.40	10.00
60 0476	F	126.0	7.5	I	46.20	46.20	4.00	10.90
40 6694	F	129.5	7.0	II	60.89	60.89	4.00	10.90
40 3407	F	126.6	8.1	I	46.20	46.20	4.40	10.90
38 4338	F	126.4	8.7	I	46.20	46.20	4.40	12.00
44 2511	F	130.0	8.0	I	46.20	46.20	4.00	10.90
94 7230	F	125.7	8.0	I	46.20	46.20	4.40	12.00
66 8507	M	127.8	8.0	II	75.59	75.59	4.40	10.00
50 4079	M	125.5	8.6	II	60.89	60.89	5.00	10.00
54 3185	M	125.0	8.0	I	46.20	46.20	4.40	10.90
62 5824	M	125.8	7.8	II	60.89	60.89	4.40	10.00
82 4044	M	127.4	7.8	II	60.89	60.89	4.40	10.00
66 4731	F	127.0	8.2	I	46.20	46.20	4.40	12.00
52 4724	F	128.0	8.0	I	46.20	46.20	4.00	10.00
60 3008	M	127.0	8.1	I	46.20	46.20	4.40	10.90
50 1729	F	125.0	8.0	I	46.20	46.20	4.40	12.00
46 9704	F	127.4	6.0	II	60.89	60.89	4.40	10.00
MEAN (x)							4.35	10.76
STANDARD DEV (s)							0.22	0.76
STD. ERROR							0.05	0.17
S.E. % OF MEAN							1.15	1.58
N							20	20
UPPER 5% t DIST.							2.0930	1.7291
A VALUES							1.0E-01	2.9E-01
LABEL SPF							4	10

MED: Minimal Erythema Dose
 I: Intensity of light source

Evaluation Period:

This study was conducted from August 7, 2000
 through August 22, 2000.



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EVALUATION OF SUN PROTECTION
BY SPF DETERMINATION (FDA-TFM)- STATIC

AMA Ref. No.: MS00.SPF.C1171TK.ST20

Date: August 22, 2000

Sponsor: Tri-K Industries
151 Veterans Drive
Northvale, NJ 07647-0128

1.0 Objective:

This panel has been convened to evaluate the effectiveness of a test material as a sunscreen product by determining the Sun Protection Factor (SPF) on human skin in a procedure defined by the Tentative Final Monograph, "Sunscreen Drug Products For Over-The-Counter Human Drugs", (Federal Register Volume 58, Number 90 pages 28194-28302, 1993) using a Xenon arc solar simulator as the UV source.

2.0 Sample Description:

On August 7, 2000 one test sample labeled Sunscreen Lotion EK4-95B was received from Tri-K Industries and assigned AMA Lab No. C-1171.

3.0 Test Material Handling:

Upon arrival at AMA Laboratories, Inc., the test material is assigned a unique laboratory code number and entered into a daily log identifying the lot number, sample description, sponsor, date received and tests requested.

Samples are retained for a period of three months beyond submission of final report unless otherwise specified by the sponsor or if sample is known to be in support of governmental applications, in which case retained samples are kept two years beyond final report submission.

Sample disposition is conducted in compliance with appropriate federal, state and local ordinances.

4.0 Panel Demographics:

Number of subjects enrolled.....	20
Number of subjects completing study.....	20
Age Range.....	18 – 52
Sex.....	Male..... 9
	Female..... 11
Race.....	Caucasian..... 20
	Hispanic..... 0
	Asian..... 0

4.1 Standards For Inclusion In A Study:

- a. Individuals between the ages of 18 and 55.
- b. Individuals free of any dermatological or systemic disorder which would interfere with the results, at the discretion of the Investigator.
- c. Individuals free of any acute or chronic disease that might interfere with or increase the risk of study participation.
- d. Individuals with Fitzpatrick's skin type I, II, and III only.
- e. Individuals with no uneven skin tones, pigmentation, scars, other irregularities or hair in test site areas that would interfere with SPF determination.
- f. Individuals who will complete a preliminary medical history form mandated by AMA Laboratories, Inc. and are in general good health.
- g. Individuals who will read, understand and sign an informed consent document relating to the specific type of study they are subscribing. Consent forms are kept on file and are available for examination on the premises of AMA Laboratories, Inc. only.
- h. Individuals able to cooperate with the Investigator and research staff, be willing to have test materials applied according to the protocol, and complete the full course of the study.
- i. Individuals willing to refrain from using any sunscreen products, sunbathing or tanning bed use, 24 hours prior to study initiation and the entire duration of the study.

- j. Individuals with excessive hair on their back who are willing to clip or shave their hair.

4.2 Standards Of Exclusion From The Study:

- a. Individuals who are under doctor's care.
- b. Individuals who are currently taking any medication (topical or systemic) that may mask or interfere with the test results.
- c. Subjects with a history of any form of skin cancer, melanoma, lupus, psoriasis, connective tissue disease, diabetes or any disease that would increase the risk associated with study participation.
- d. Individuals diagnosed with chronic skin allergies.
- e. Individuals with a history of adverse effects upon sun exposure.
- f. Female volunteers who indicate that they are pregnant or nursing.
- g. Individuals with blemishes, nevi, sunburn, suntan, scars, moles, active dermal lesions or uneven pigmentation in the test sites.
- h. Individuals with known hypersensitivity to any sunscreen products.

4.3 Informed Consent And Medical History Forms:

An informed consent was obtained from each volunteer prior to initiating the study describing reasons for the study, possible adverse effects, associated risks and potential benefits of the treatment and their limits of liability. Panelists signed and dated the informed consent document to indicate their authorization to proceed and acknowledge their understanding of the contents. Each subject was assigned a permanent identification number and completed an extensive medical history form. These forms along with the signed consent forms are available for inspection on the premises of AMA Laboratories, Inc. only. Reference 21 CFR Ch. 1 Part 50, Subpart B.

A trained technician performed a physical examination of the panelist's back to determine if study eligibility criteria were satisfied.

4.4 Panel Composition:

Healthy volunteers over the age of 18 years were recruited for this study. The panel consisted of fair-skin individuals with skin types I, II or III defined as follows (Federal Register Vol. 58, No. 90: 28299, 1993).*

Type I - Always burns easily; never tans

Type II - Always burns easily; tans minimally

Type III - Burns moderately; tans gradually

* - Based on the first 30 to 45 minutes sun exposure after a winter season of no sun exposure.

4.5 Institutional Review Board:

Reference: CFR Title 21 Part 56, Subparts A, B, C, and D. The IRB of AMA Laboratories, Inc., consists of five or more individuals, chosen from within the company for technical expertise and from the local community for lay interaction. The list of IRB members is kept on file at AMA Laboratories, Inc. and is available for inspection during the hours of operation.

5.0 Artificial Light Source:

The light source employed is a 150 watt Xenon Arc Solar Simulator (Solar Light Co., Philadelphia, Pennsylvania, Model 12S, Model 14S or Model 600) having a continuous emission spectrum in the UVB range from 290 to 320 nm. Xenon arc is selected on the basis of its black body radiation temperature of 6000° K which produces continuous UV spectra (all wavelengths) substantially equivalent to that of natural sunlight¹.

This device is equipped with a dichroic mirror (which reflects all radiation below 400nm) and works in conjunction with a 1 mm thick Schott WG-320 filter (which absorbs all radiation below 290nm) to produce simulation of the solar UVA-UVB spectrum. A 1 mm thick UG 11 filter (black lens) was added to remove reflected (infra-red, greater than 700 nm) heat and remaining visible radiation. UVB radiation was monitored continuously during exposure using a Model DCS-1 Sunburn UV Meter/Dose Controller System (Solar Light Co.) formerly known as the Robertson-Berger Sunburn Meter

(R-B meter). Measurements were taken at a position within 8 mm from the surface of the skin. The field of irradiation was 1 cm in diameter. The solar simulator was allowed a warm up time of at least 15 minutes before use and power supply output was recorded.

Realignment of the Light Sources and calibration of the sunburn meters are conducted semi-annually by independent certification facilities and more often as necessary at the discretion of the operating technician or Investigator.

1/ Berger, D.S.: Specification and Design of Solar Ultraviolet Simulators. J. Invest. Dermatol. 53: 192-199, 1969.

6.0 Procedure:

STATIC SPF DETERMINATION (INCLUDING 8% HOMOSALATE STANDARD)

The infrascapular area of the back to the right and left of the midline was used. Within this area, 50 cm² rectangular test sites were delineated with a gentian violet surgical skin marker. Sites were observed to ensure uniform pigmentation, skin tone and texture, and absence of warts, moles, nevi, scars, blemishes and active dermal lesions. Any areas that might be expected to produce erratic results were not used for UV exposures.

The procedure for this study is outlined in the Federal Register, Vol. 58: No.90, 28194-28302. One test site area served to determine each subject's Minimal Erythema Dose (MED). A minimum of five UV exposures was administered within this site. The individual subject's MED is the shortest time of exposure that produces minimally perceptible erythema at 22 to 24 hours post irradiation.

The test material and 8% homosalate standard were shaken and/or swirled with a glass rod before use and were evenly applied using plastic volumetric syringes to a rectangular areas measuring 5 cm x 10 cm (50 cm²) for a final concentration of 2.0 mg/cm². Evenness of application was verified by observation with a Woods Lamp.

Fifteen minutes after application, a protected site received a series of seven UV exposures based upon previously determined MED.

The UV exposures for test products and 8% homosalate standard were calculated from previously determined MED and the intended SPF as follows:

SPF 4: MED times 0.64x, 0.80x, 0.90x, 1.00x, 1.10x, 1.25x and 1.56x

SPF 15: MED times 0.76x, 0.87x, 0.93x, 1.00x, 1.07x, 1.15x and 1.32x where x equals the expected SPF of the product.

7.0 Evaluation Of Responses:

Twenty-two to twenty-four hours post exposure, the subjects are instructed to return to the testing facility for evaluation of delayed erythemic responses. The technician who evaluates the MED did not know the identity of the test product application sites and UV exposures. Also he/she was not the same person to have applied the sunscreen product to the test site or administered the doses of UV radiation.

$$\text{SPF} = \frac{\text{Protected MED}}{\text{Final unprotected MED}}$$

Visual grading scale:

- 0 = No Erythema
- ? = Questionable Erythema
- 1 = Minimal Erythema
- 2 = Slight Erythema
- 3 = Well-Defined Erythema
- 4 = Erythema and Edema
- 5 = Erythema and Edema in vesicles

- 7.1 All technical employees of AMA Laboratories, Inc. are required to take and pass a visual discrimination examination conducted by a Board Certified Ophthalmologist using the Farnsworth-Munsell 100 Hue Test as published; which determines a person's ability to discern color against a black background. This test was additionally modified to include a flesh tone background more nearly approaching actual use conditions, wherein erythematous skin is graded according to intensity.

8.0 Determination of the Test Product's SPF Value and PCD:

Calculation of SPF and PCD – The mean SPF value (x) was calculated. The standard deviation was determined (s). The upper 5% point was obtained from the t distribution table with n-1 degrees of freedom (t). First, A was calculated as follows:

$$A = \frac{(t)(s)}{\sqrt{n}}$$

Therefore, the label SPF for panels using a minimum of 20 evaluable subjects is the largest whole number less than the mean SPF minus A.

$$\text{Label SPF} = \text{Mean SPF} - A$$

The Product Category Designation (PCD), for labeling purposes, was assigned based on the mean SPF and PCD ranking according to the TFM. Classifications may be Ultra High, Very High, High, Moderate or Minimal.

9.0 Rejection Criteria:

Panelist's results were rejected and the panelist replaced if:

1. An exposure series fails to elicit an MED response on the untreated skin. The test is considered a technical failure even if the MED response is observed in the protected site.
2. The responses on the protected area are randomly absent, indicating uneven product spreading, non-constant light irradiance or unstable product.
3. All exposures in a series elicit responses - thus prohibiting any MED calculation.

10.0 Results: Please see attached Table.

11.0 Observations:


No adverse effects or unexpected reactions of any kind were observed on any of the subjects.

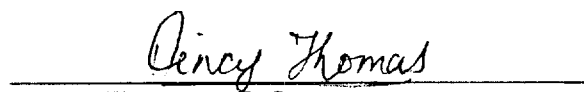
12.0 Archiving:

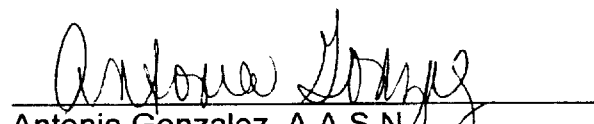
All original samples, raw data sheets, technicians notebooks, correspondence files and copies of final reports and remaining specimens are maintained on premises of AMA Laboratories, Inc. in limited access storage files marked "Archive". A duplicate disk copy of final reports is separately archived in a bank safe deposit vault.

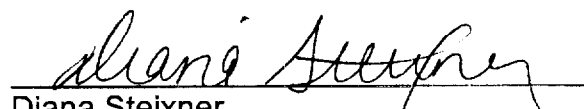
13.0 Conclusions:

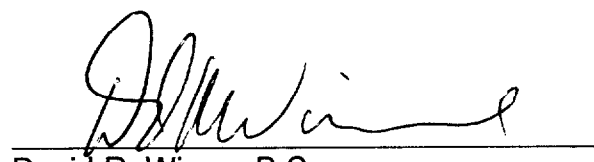
The Sun Protection Factor (SPF) of the above test material (AMA Lab No.: C-1171; Client No.: Sunscreen Lotion EK4-95B) when tested on twenty subject as described herein under static conditions yielded the mean SPF value of 18.31, which can be assigned a Products Category Designation (PCD) of Very High according to the reference. The mean SPF of the 8% homosalate standard on the same panel was 4.35

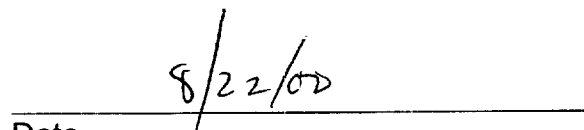

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Table

Sponsor: Tri-K Industries
 AMA Lab No.: C-1171
 Client No.: Sunscreen Lotion EK4-95B

Subject ID #	Sex	MED/HR	I (Amps)	Skin Type	MED I J/M ²	MED II J/M ²	STD (8% HMS)	SPF Value
36 2460	F	126.7	8.0	I	46.20	46.20	4.40	18.00
36 3821	M	127.0	7.0	II	60.89	60.89	4.40	18.00
64 1135	M	125.6	8.1	II	75.59	75.59	4.40	18.00
68 6947	M	128.3	9.0	II	60.89	60.89	4.40	16.35
60 0476	F	126.0	7.5	I	46.20	46.20	4.00	21.60
40 6694	F	129.5	7.0	II	60.89	60.89	4.00	16.35
40 3407	F	126.6	8.1	I	46.20	46.20	4.40	18.00
38 4338	F	126.4	8.7	I	46.20	46.20	4.40	21.60
44 2511	F	130.0	8.0	I	46.20	46.20	4.00	18.00
94 7230	F	125.7	8.0	I	46.20	46.20	4.40	21.60
66 8507	M	127.8	8.0	II	75.59	75.59	4.40	18.00
50 4079	M	125.5	8.6	II	60.89	60.89	5.00	18.00
54 3185	M	125.0	8.0	I	46.20	46.20	4.40	18.00
62 5824	M	125.8	7.8	II	60.89	60.89	4.40	16.35
82 4044	M	127.4	7.8	II	60.89	60.89	4.40	18.00
66 4731	F	127.0	8.2	I	46.20	46.20	4.40	21.60
52 4724	F	128.0	8.0	I	46.20	46.20	4.00	16.35
60 3008	M	127.0	8.1	I	46.20	46.20	4.40	18.00
50 1729	F	125.0	8.0	I	46.20	46.20	4.40	16.35
46 9704	F	127.4	6.0	II	60.89	60.89	4.40	18.00
MEAN (x)							4.35	18.31
STANDARD DEV (s)							0.22	1.83
STD. ERROR							0.05	0.41
S.E. % OF MEAN							1.15	2.24
N							20	20
UPPER 5% t DIST.							2.0930	1.7291
A VALUES							1.0E-01	7.1E-01
LABEL SPF							4	18

MED: Minimal Erythematous Dose
 I: Intensity of light source

Evaluation Period:

This study was conducted from August 7, 2000
 through August 22, 2000.



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EVALUATION OF UVA-PROTECTION FACTOR

AMA Ref. No.: MS00.UVA.PPDC1168TK.J10
Date: August 29, 2000
Sponsor: Tri-K Industries
151 Veterans Drive
Northvale, New Jersey 07647-0128

1.0 Objective:

This panel has been convened to determine UVA protection afforded by a sunscreen product using the Persistent Pigment Darkening (PPD) response. (Japan Cosmetic Industry Association - J.C.I.A.- Measurement Standards for UVA Protection Efficacy).

2.0 Sample Description:

On August 7, 2000 four test samples labeled as listed below were received from Tri-K Industries and assigned AMA Lab Nos. as follows:

Sample Description	AMA Lab No.
Sunscreen Lotion EK4 96A	C-1168
Sunscreen Lotion EK4 96C	C-1169
Sunscreen Lotion EK4 95E	C-1170
Sunscreen Lotion EK4 95B	C-1171

3.0 Test Material Handling:

Upon arrival at AMA Laboratories, Inc., the test material is assigned a unique laboratory code number and entered into a daily log identifying the lot number, sample description, sponsor, date received and tests requested.

Samples are retained for a period of no less than two years beyond submission of final report.

Sample disposition is conducted in compliance with appropriate federal, state and local ordinances.

Data are compiled onto floppy disk files. Back up disks are stored in a bank vault for safe keeping and retrieval in the event of hazard loss.

4.0 Panel Demographics:

Number of subjects enrolled.....	10
Number of subjects completing study.....	10
Age Range.....	19 – 49
Sex.....	Male..... 7
	Female..... 3
Race.....	Caucasian..... 10
	Hispanic..... 0
	Asian..... 0

4.1 Standards For Inclusion In A Study:

1. Individuals who are at least 18 years of age, in good general health who are not currently under a physician's care for any medical condition.
2. Individuals with self-reported Fitzpatrick Skin Types II, III or IV.
3. Individuals with no uneven skin tones, pigmentation, scars or other irregularities in the treatment area.
4. Individuals willing to refrain from using other topical products on the back during the study.
5. Individuals willing to refrain from taking anti-inflammatory drugs (e.g., aspirin, ibuprofen) and other medications during the study without prior approval of the Investigator.
6. Individuals willing to return to the study site for treatment/evaluation at specified times during the study.
7. Individuals who give written informed consent by signing the Informed Consent Form provided by the Investigator.
8. Individuals willing to refrain from using commercial sunscreen products, have sun exposure or visit a tanning salon 24 hours prior to study initiation and during the study.

4.2 Standards For Exclusion From A Study:

1. Individuals who are or suspect that they are pregnant or who are lactating will be excluded on the basis of possible altered efficacy response to ultraviolet light.

2. Individuals with the presence or history of skin cancer(s).
3. Individuals with a history of toxic or allergic responses to sun exposure or photo- sensitive skin disease(s).
4. Individuals who have been diagnosed by a physician as having atopy, psoriasis, eczema, or other chronic skin diseases.
5. Individuals who exhibited hypersensitivity, rash or other abnormal skin reactions or lesions to topical or systemic medications, sunscreens, cosmetics or fragrances in the 7 days prior to study initiation.
6. Individuals who have been treated in the last 14 days with any medication(s) that can change the body's response to ultra- violet radiation. These include but are not limited to thiazides, phenothiazines, anti-biotics, corticosteroids, antihistamines and anti-inflammatory drugs (e.g., aspirin, ibuprofen).
7. Individuals with obvious recent observable exposure to sunlight or solar simulated light as evidenced by discernible erythema and/or tanning.
8. Individuals with Fitzpatrick Skin Types I, V and VI.
9. Individuals with known hypersensitivity to any sunscreen agents.
10. Individuals with other conditions considered by the Investigator as sound reasons for disqualification from enrollment into the study.

4.3 Informed Consent And Medical History Forms:

An informed consent was obtained from each volunteer prior to initiating the study describing reasons for the study, possible adverse effects, associated risks and potential benefits of the treatment and their limits of liability. Panelists sign and date the informed consent document to indicate their authorization to proceed and acknowledge their understanding of the contents. Each subject was assigned a permanent identification number and completed an extensive medical history form. These forms along with the signed consent forms are available for inspection on the premises of AMA Laboratories, Inc. only. Reference 21 CFR Ch. 1 Part 50, Subpart B.

4.4 Panel Composition:

Normal, healthy, adult volunteers who are 18 through 60 years of age were recruited for this study. The panel

consisted of individuals with skin types II, III and IV defined as follows (Federal Register 43: 38260, 1978).

Type II - always burns easily; tans minimally (sensitive)

Type III - burns moderately; tans gradually

Type IV - burns minimally; always tans well

5.0 Light Source:

The light source employed is a 150 watt Xenon Arc Solar Simulator¹ (Solar Light Co., Philadelphia, Pennsylvania, Model 12S, 14S or Model 600) equipped with an Ultraviolet(UV) reflecting dichroic mirror, 3mm thick Schott WG-335 filter together with a 1mm thick Schott UG-11 filter was used to produce simulation of the UVA solar spectrum.

UVA radiation was monitored continuously during exposure using a Model DCS-1 Sunburn UV Meter/Dose Controller System (Solar Light Co.) formerly known as the Robertson-Berger Sunburn meter (R-B meter). Measurements were taken at a position within 8 mm from the surface of the skin. The field of irradiation was 1 cm in diameter. Realignment of the Light Sources and calibration of the sunburn meters are conducted semi-annually by independent certification facilities and more often as necessary at the discretion of the operating technician.

1/ Berger, D.S.: Specification and design of solar ultraviolet simulators. J. Invest. Dermatol. 53: 192-199, 1969.

6.0 Procedure:

TREATMENT AND EXPOSURE AREAS

The mid to lower untanned back, lateral to the midline, was used for the treatment and exposure areas. A minimum of 20 cm² test product areas were delineated with a surgical skin marker.

MPPD DETERMINATION

The threshold dose for PPD in unprotected skin was determined over the mid to lower back by administering a series of exposures in 25% dose increments of UVA radiation in geometric progression. The Minimum PPD dose (MPPD) is the smallest UVA dose required to produce PPD 2 to 4 hours after exposure. A minimum of 5 exposures were made. The MPPD of unprotected skin was determined under standardized lighting conditions 2 to 4 hours after

exposures. The threshold response was taken as an unequivocal pigment darkening with distinct borders which persisted for at least 2 to 4 hours.

Since MPPD is determined 2 to 4 hours after exposures, there should be little or no risk of failing to elicit a threshold dose. Persistent pigmentation on each subsite was graded according to the following 4 point ordinal scale:

- 0 = No discernable pigment darkening
- +/- = Barely perceptible pigment
- 1 = Unequivocal pigment darkening, distinct borders, lasting more than 2 to 4 hours
- 2 = Pronounced pigment darkening, lasting more than 2 to 4 hours

TEST MATERIAL APPLICATION AND UVA EXPOSURE

One hundred milligrams of test material and JCIA Standard were applied to the test sites through plastic tuberculin syringes. The test material was evenly applied to an area of at least 20cm² for a final concentration of 2.0 mg/cm² or 2 ul/cm². Fifteen minutes after application, a series of UVA light exposures were administered at 25% increments. Test areas were far enough apart that the solar simulator patient stop did not touch the next exposure area.

7.0 Evaluation Of Responses

The threshold PPD within each site was determined according to the 4 point ordinal scale stated in section 6.0.

The UVA - Protection Factor(UVA-PF) was calculated as follows:

$$\text{UVA-PF} = \frac{\text{MPPD(Seconds)} - \text{Protected skin}}{\text{MPPD(Seconds)} - \text{Unprotected skin}}$$

7.1

All technical employees of AMA Laboratories, Inc. are required to take and pass a visual discrimination examination conducted by a Board Certified Ophthalmologist using the Farnsworth-Munsell 100 Hue Test as published; which determines a person's ability to discern color against a black background. This test was additionally modified to include a flesh tone background more nearly approaching actual use conditions, wherein erythematous skin is graded according to intensity.

8.0 Rejection Criteria:

Panelist's results were rejected and the panelist replaced if:

1. The responses on the treated test site were randomly absent or out of sequence. This was an indication that the products were not spread uniformly.
2. If an MPPD was not produced at an exposure equivalent to 6.0 J/cm², the subject most likely did not show a PPD in response to UVA and was discontinued and another subject substituted.
3. The UV exposure series, for either the unprotected (untreated) or protected (treated) sites, failed to show a distinct MPPD where all sites scored less than a 1 or 2 or greater. In such a case, a unique UVA-PF cannot be determined.

9.0 Adverse Reactions:

Subjects were instructed to promptly report adverse effects to the Investigator. The Investigator would then determine the need for interim examination and, if warranted, termination from the study. Any adverse effect(s), spontaneously expressed by the subject or observed by the Investigator, during or after the study were recorded on an Adverse Effect(s)/Intercurrent Event(s) Report.

10.0 Results:

Please see attached Tables.

11.0 Observations:

No adverse effects or unexpected reactions of any kind were observed on any of the subjects.


12.0 Archiving:


All original samples, raw data sheets, technicians notebooks, correspondence files and copies of final reports and remaining specimens are maintained on premises of AMA Laboratories in limited access storage files marked "Archive". A duplicate disk copy of final reports is separately archived in a bank safe-deposit vault.

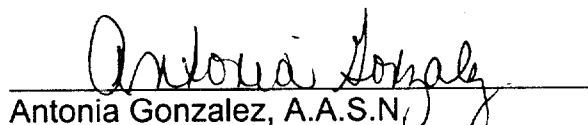
13.0 Conclusions: The mean UVA Protection Factor (UVA-PF) of the samples listed below when tested with a WG-335 filter according to the reference described herein were:

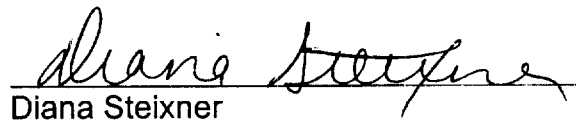
AMA Lab No.	Sample Description	UVA-PF
C-1168	Sunscreen Lotion EK4 96A	4.58
C-1169	Sunscreen Lotion EK4 96C	8.80
C-1170	Sunscreen Lotion EK4 95E	4.79
C-1171	Sunscreen Lotion EK4 95B	9.17


The mean UVA Protection Factor of the JCIA standard on the same panel was 4.89.


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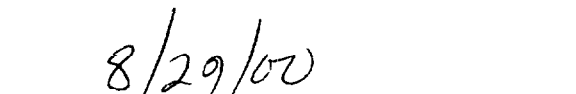

Date

Table 1

Sponsor: Tri-K Industries
 AMA Lab No.: C-1168
 Client No.: Sunscreen Lotion EK4 96A

Subject ID #	Sex	MW/cm2	A	Skin Type	MPPD	MPPD II	JCIA STD	UVA-PF
56 9719	M	68.0	7.4	III	81	81	4.77	4.77
60 5763	F	69.7	7.5	III	65	65	5.94	3.80
62 5824	M	70.1	7.5	III	81	81	4.77	3.81
54 4669	F	65.0	7.5	II	65	65	4.75	4.75
52 2152	M	71.4	8.0	III	81	81	4.77	4.77
40 6694	F	68.4	6.0	III	81	81	4.77	4.77
74 8993	M	62.0	8.0	III	81	81	4.77	4.77
66 8507	M	68.9	8.0	III	81	81	4.77	4.77
74 5942	M	70.0	6.5	III	81	81	4.77	4.77
70 0795	M	68.6	6.0	III	101	101	4.78	4.78
MEAN UVA-PF							4.89	4.58
STANDARD DEV							0.37	0.41
STD. ERROR							0.12	0.13
S.E. % OF MEAN							2.45	2.84
N OF CASES							10	10

Evaluation Period: This study was conducted from August 7, 2000 through August 25, 2000.

Table 2

Sponsor: Tri-K Industries
 AMA Lab No.: C-1169
 Client No.: Sunscreen Lotion EK4 96C

Subject ID #	Sex	MW/ Cm2	A	Skin Type	MPPD	MPPD II	JCIA STD	UVA-PF
56 9719	M	68.0	7.4	III	81	81	4.77	9.31
60 5763	F	69.7	7.5	III	65	65	5.94	7.43
62 5824	M	70.1	7.5	III	81	81	4.77	9.31
54 4669	F	65.0	7.5	II	65	65	4.75	7.43
52 2152	M	71.4	8.0	III	81	81	4.77	9.31
40 6694	F	68.4	6.0	III	81	81	4.77	11.64
74 8993	M	62.0	8.0	III	81	81	4.77	9.31
66 8507	M	68.9	8.0	III	81	81	4.77	9.31
74 5942	M	70.0	6.5	III	81	81	4.77	7.44
70 0795	M	68.6	6.0	III	101	101	4.78	7.47
MEAN UVA-PF							4.89	8.80
STANDARD DEV							0.37	1.36
STD. ERROR							0.12	0.43
S.E. % OF MEAN							2.45	4.89
N OF CASES							10	10

Evaluation Period: This study was conducted from August 7, 2000 through August 25, 2000.

Table 3

Sponsor: Tri-K Industries
 AMA Lab No.: C-1170
 Client No.: Sunscreen Lotion EK4 95E

Subject ID #	Sex	MW/cm2	A	Skin Type	MPPD	MPPD II	JCIA STD	UVA-PF
56 9719	M	68.0	7.4	III	81	81	4.77	4.77
60 5763	F	69.7	7.5	III	65	65	5.94	3.80
62 5824	M	70.1	7.5	III	81	81	4.77	4.77
54 4669	F	65.0	7.5	II	65	65	4.75	4.75
52 2152	M	71.4	8.0	III	81	81	4.77	5.96
40 6694	F	68.4	6.0	III	81	81	4.77	4.77
74 8993	M	62.0	8.0	III	81	81	4.77	4.77
66 8507	M	68.9	8.0	III	81	81	4.77	4.77
74 5942	M	70.0	6.5	III	81	81	4.77	4.77
70 0795	M	68.6	6.0	III	101	101	4.78	4.78
MEAN UVA-PF							4.89	4.79
STANDARD DEV							0.37	0.51
STD. ERROR							0.12	0.16
S.E. % OF MEAN							2.45	3.34
N OF CASES							10	10

Evaluation Period: This study was conducted from August 7, 2000 through August 25, 2000.

Table 4

Sponsor: Tri-K Industries
 AMA Lab No.: C-1171
 Client No.: Sunscreen Lotion EK4 95B

Subject ID #	Sex	MW/cm2	A	Skin Type	MPPD	MPPD II	JCIA STD	UVA-PF
56 9719	M	68.0	7.4	III	81	81	4.77	9.31
60 5763	F	69.7	7.5	III	65	65	5.94	7.43
62 5824	M	70.1	7.5	III	81	81	4.77	11.64
54 4669	F	65.0	7.5	II	65	65	4.75	9.28
52 2152	M	71.4	8.0	III	81	81	4.77	9.31
40 6694	F	68.4	6.0	III	81	81	4.77	9.31
74 8993	M	62.0	8.0	III	81	81	4.77	9.31
66 8507	M	68.9	8.0	III	81	81	4.77	9.31
74 5942	M	70.0	6.5	III	81	81	4.77	7.44
70 0795	M	68.6	6.0	III	101	101	4.78	9.34
MEAN UVA-PF							4.89	9.17
STANDARD DEV							0.37	1.17
STD. ERROR							0.12	0.37
S.E. % OF MEAN							2.45	4.04
N OF CASES							10	10

Evaluation Period: This study was conducted from August 7, 2000 through August 25, 2000.